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GASES IN THE COMMERCIAL HANDLING OF CITRUS JUICES

by

George N. Pulley and Harry W. von Loesecke
U. S. Citrus Products Station, Bureau of Agricultural Chemistry & Engineering,
U. S. Department of Agriculture, Winter Haven, Fla.

A study has been made of the gases present in Florida citrus juices (grapefruit and orange) extracted and deaerated by different methods in the laboratory and in commercial practice.

The oxygen content of commercially extracted citrus juices before deaeration ranged from 2.46 to 4.67 cc per liter, reduced to standard conditions. After deaeration they contained 0.09 to 2.39 cc per liter. The commercial plate-type deaerators investigated were found to be greatly overloaded with a resulting reduction in efficiency. Under the conditions of the investigations, centrifugal deaeration was found to be the most efficient of those methods examined and now being used in commercial practice in Florida. Using this type of deaerator, most efficient deaeration is obtained when the temperature of the juice is not less than 61°F. (16°C.) with a vacuum of at least 25 inches (635 mm.).

Dissolved oxygen in the juice apparently reacts with some constituent or constituents of the juice and results in a disappearance of the oxygen. This disappearance of oxygen is accelerated by elevated temperatures and may adversely affect the juice, especially so far as the vitamin C content is concerned.

Because oxidation is believed to be one of the causes of off-flavors in canned citrus juices (mainly orange and grapefruit), the present work was undertaken to learn the quality of oxygen present in such juices when prepared by different methods of extraction and deaeration. Another purpose of the present paper is to show the efficiency of various methods of deaeration as now being used in some Florida citrus canneries. Further work is contemplated relative to the gas present in canned citrus juices stored for different periods of time.

Published data on the gas content of citrus juices are meager. Lachele (3) mentions the determination of gases in fruit and vegetable juices but does not give complete data. Eddy (1) found that the absorption of oxygen by orange juice caused a reduction in vitamin C content. Toulouse (6) reports that oxygen in carbonated beverages is gradually consumed and may affect the flavor of the product. He also found that oxygen consumption is more rapid in beverages containing citrus fruits and causes them to develop a bitter flavor.

Apparatus for Determining Gas

The apparatus used for determining the gas content of the juice was an adaptation of the modified Peterson equipment (7) to which was attached a Sprengel pump (Figure 1):

To operate the apparatus, stopcocks 3 and 4 are opened, and about 20 cc. of

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the acidified salt solution in the gas buret E are drawn into mercury pump C by lowering leveling bulb D. This salt solution consists of 200 grams of anhydrous sodium sulfate, 800 cc of distilled water, and 40 cc of concentrated sulfuric acid (2). Stopcock 3 is then closed. Bulb D is raised and stopcock 3 opened to connect with I in order to sweep out all gas. Tube I should be completely filled with the acidified salt solution. In carrying out the above manipulation, buret E and connecting tube G, as well as the space above the mercury in C, should be completely filled with the acidified salt solution. Stopcock 1 is opened and the height of leveling bulb A adjusted so that the mercury will be even with the bottom of flask B, which contains the sample to be analyzed. The volume of sample taken should be about 75 cc. less than the capacity of flask B and introduced in the flask by siphoning to avoid aeration. Stopper J should be shellacked to avoid all leaks. Stopcock 2 is now opened (stopcock 3 should be closed), and leveling bulb A raised until the sample of juice completely fills tube H. Then stopcock 2 is closed and bulb A lowered until it is 762 mm. (30 inches) below stopcock 1, but none of the sample should be allowed to pass below stopcock 1. Stopcock 1 is then closed. The sample in flask B will now be under a high vacuum and there will be an evolution of gas. Bulb D is lowered until it is 762 mm below point p on mercury pump C, and then stopcock 3 is carefully opened so that flask B is connected with mercury pump C. If the liquid in B foams violently when this is done, stopcock 3 is closed and flask B gently tapped to settle the foam. Then stopcock 3 is again carefully opened. When violent foaming has ceased, mercury pump C and flask B are allowed to remain connected, and the water bath containing flask B is gently warmed. The juice is not warmed until most of the gas has been collected from the juice while still at room temperature. The purpose of this is to avoid as much as possible any combination of the gas with the constituents of the juice. After an interval of one minute, stopcock 3 is closed and bulb D raised. Stopcock 3 is opened so that the gas collected will be forced into buret E. Bulb D is now lowered and stopcock 3 opened so that mercury pump C and flask B are again connected. After another minute the gas is transferred to buret E as above. This operation is repeated until less than 0.5 cc. of gas is collected in a 5-minute interval. When this stage has been reached, it is assumed that for all practical purposes the gas has been exhausted from the sample. In the case of fresh orange and grapefruit juices, 1.5 hours are necessary to reach this point.

The temperature of the water in the bath should not exceed 55-60°C. (131-140°F).

The volume of gas in buret E (which will amount to 28-48 cc for a 800 cc sample of orange or grapefruit juice) is recorded along with the temperature of the water jacket surrounding buret E and the barometer reading. The gas in buret E is then run into absorption pipets for the determination of carbon dioxide and oxygen.

An oxygen blank was established by determining the oxygen in water by means of the apparatus and comparing this with the oxygen obtained by the Winkler method. For this apparatus the blank was found to be 0.23 cc of oxygen per liter and was subtracted from all oxygen readings obtained.

It is not possible to make reliable duplicate determinations on the same sample because, while one determination is being carried out, the oxygen will be slowly consumed in the juice as it stands awaiting the second determination. As an example, a sample of orange juice was stored in a tightly stoppered flask. The first determination showed 3.4 cc of oxygen per liter; the second determination 2 hours later showed 2.8 cc of oxygen per liter.

In carrying out the determinations, practically all the oxygen and nitrogen in the juice is immediately removed at a low (room) temperature, and the last traces are removed by heating the juice to about 55° C. It would seem, therefore, that the oxygen in the sample has little time to react with the constituents of the juice and thus incorporate a significant error.

Gas Content of Citrus Juice

The gas content of citrus juices extracted by different methods is shown in Table 1. After the juice had been extracted by the methods indicated, it was either passed through a finisher or through vibrating screens. A considerable amount of air was probably beaten into the juice by this screening. All samples were analyzed immediately after collection.

Table I. Gas Content of Citrus Juices Extracted by Different Methods

Method	Tot.Gas	CO_2	O_2	N_2	Ratio O_2/N_2
	cc/liter at 0° C. (760 mm)				
Rapid reaming:					
Lab. scale (hand)	48.2	31.1	3.47	13.6	0.258
Plant operation	28.5	14.7	4.02	9.7	0.415
Plant operation	42.8	28.2	3.24	11.3	0.287
Rasping and pressing (Plant operation)	36.7	22.3	4.17	10.2	0.409
Slow reaming (lab. scale)	54.9	38.7	2.52	13.7	0.184
Peeling and screw press (lab. scale)	57.7	41.5	2.22	13.9	0.160
Mechanical (plant operation)	35.5	22.3	2.46	10.7	0.230

The technique of rapid reaming consists of cutting the fruit in half and then pressing each half by hand over rapidly revolving ribbed burrs. These burrs revolve at about 1750 r.p.m.; in slow reaming they revolve at 70 r.p.m. In the method of screw pressing, the fruit is first peeled and then fed into a screw surrounded by a screen. The mechanical extractor used was operated by first halving and then quartering the fruit while two burrs, revolving at different speeds, reamed the interior of the quarters. Juice was also extracted by rasping the fruit to remove the flavedo, cutting in half, and then pressing the halves in a suitable type of continuous press.

Comparisons of the amount of oxygen in slow, rapid-reamed, and screw-press juice (Table I) can be made only in laboratory-scale operations because in these cases the extracted juice was screened in exactly the same manner. If these results are compared, it will be seen that rapid reaming incorporates the most oxygen. In the case of plant operations, the juice was screened in different ways in each of the plants, and therefore data for plant operations cannot be compared.

The ratio of oxygen to nitrogen in the extracted juice is not the same as that in air, nor does there seem to be any consistency in this ratio for extracted juice. Furthermore, the ratio in the juice was always less than the ratio in water. This probably indicates a combination of oxygen with compounds in the juice.

Amount of Gas Removal by Deaeration

One of the methods of deaerating citrus juices is described by Mottern and von Loesecke (4); the juice is forced at high velocity through a small nozzle against the dome-shaped base of an inverted flask, where it spreads out in a thin film containing numerous bubbles. The flask is maintained under a vacuum, and the bubbles of air expand and burst.

Later this principle was put into commercial use by the construction of a commercial deaerator which consisted of a series of baffle plates mounted in a shell that could be maintained under a vacuum. The juice cascading over these baffles was thus subjected in the form of a thin film to the action of a vacuum (Figure 2A). This type is used commercially in Florida and consists of a shell about 6 feet (1.8 meters) high and 20 inches (50.8 cm.) in diameter in which baffles are mounted. During the tests reported here, the flow of juice amounted to 825 gallons (3123 liters) per hour with a vacuum of 28.5 inches (724 mm.).

Another type of deaerator is shown in Figure 2B. The juice flows over a perforated cylinder mounted in a jacket maintained under a vacuum. The shell in which the perforated cylinder is mounted is about 4 feet (122 cm.) high and 2 feet (61 cm.) in diameter. During the tests reported, flow of juice was at the rate of approximately 1000 gallons (3785 liters) per hour with a vacuum of 25 inches (635 mm.).

A third type is indicated in Figure 2C. The juice is made to flow upon a revolving saucer with perforated sides, the entire assembly being maintained under a vacuum (5). The juice is forced through the holes in the side of the saucer and impinges violently against the walls of the deaerator; the bubbles are thus broken. During the tests, flow of juice was at the rate of about 600 gallons (2271 liters) per hour with a vacuum of about 29 inches (736 mm.).

Another method of deaeration is to allow the juice to stand in a tank under a vacuum. The juice in the tank may or may not be violently agitated while being subjected to a vacuum, and the vacuum may or may not be replaced by inert gas.

The efficiency of these different methods as practiced commercially is shown in Table II. The data show that in the laboratory type equipment the method of spraying into a flask and the centrifugal deaerator are of nearly the same efficiency in removing oxygen. When the principle of the flask type was incorporated into a commercial design (Figure 2, A and B), the efficiency was greatly diminished. The reason is indicated in Table III which shows that plate-type deaerators used commercially in Florida are greatly overloaded.

Of the three commercial types of deaeration investigated, the plate type and perforated-cylinder type were about equal in efficiency. The centrifugal type was the most effective, and it is doubtful whether equipment could be constructed giving greater efficiency. The commercial centrifugal deaerator used in these commercial tests is shown in Figure 3 and consists of a stainless steel shell, 30 inches (76.2 cm.) high, and 20 inches (50.8 cm.) in diameter.

Lachele (3) found that the efficiency of commercial deaerators tested at various plants varied from 73 to 91 percent, depending upon the juice flow and general type of deaerating system. Perhaps exceptions might be taken to the results of our work by virtue of the fact that the rate of flow of juice in all three commercial deaerators was different. The results, however, represent what is being accomplished in commercial practice, and to decrease the flow of juice so that equal efficiency would be obtained for all three deaerators (if it could be done in this manner) would result in decreased production. The laboratory centrifugal deaerator had a rate of flow of 80 gallons of juice an hour; in the commercial machine, the rate of flow was 600 gallons per hour. Furthermore both machines were of equal efficiency although rate of flow was greatly different.

Efficiency of Centrifugal Deaeration

Effect of Temperature. The efficiency of deaeration at different temperatures maintaining a constant vacuum was determined on the laboratory model centrifugal deaerator. Since the efficiency of this laboratory deaerator was essentially the same as that of the commercial deaerator, the results obtained can be applied to commercial practice.

The results are shown in Table IV. Maximum efficiency in oxygen removal was obtained at temperatures not less than 61°F. (16°C.) when a vacuum of 27 inches (686 mm.) was used. Juice flow was maintained the same in all tests.

Effect of Vacuum. The temperature was maintained constant at 61°F. and the vacuum was varied. The results given in Table V indicate that at least a 25 inch (635 mm.) vacuum is necessary for the most efficient removal of oxygen. Juice flow was at the same rate as in the tests where the temperature was varied.

Gases in Citrus Juices at Various Steps of Processing

The gas analysis apparatus was taken to different canning plants, and determinations of the gases were made in the juice at different stages of processing. The results (Table VI) represent the average of two or more commercial runs. The comparatively large amount of oxygen in the canned juice of plant 4 is a little difficult to explain but possibly might be accounted for by the entrainment of air during filling of the cans. The canned juice in every case was taken from the cooling tank and analyzed immediately.

Oxygen in canned citrus juices rapidly disappears. Toulouse (6) found this to be the case in carbonated citrus beverages. The rate of disappearance of oxygen in canned citrus juices is much more rapid at higher temperatures (Table VII) and may adversely affect the juice, especially so far as vitamin C is concerned.

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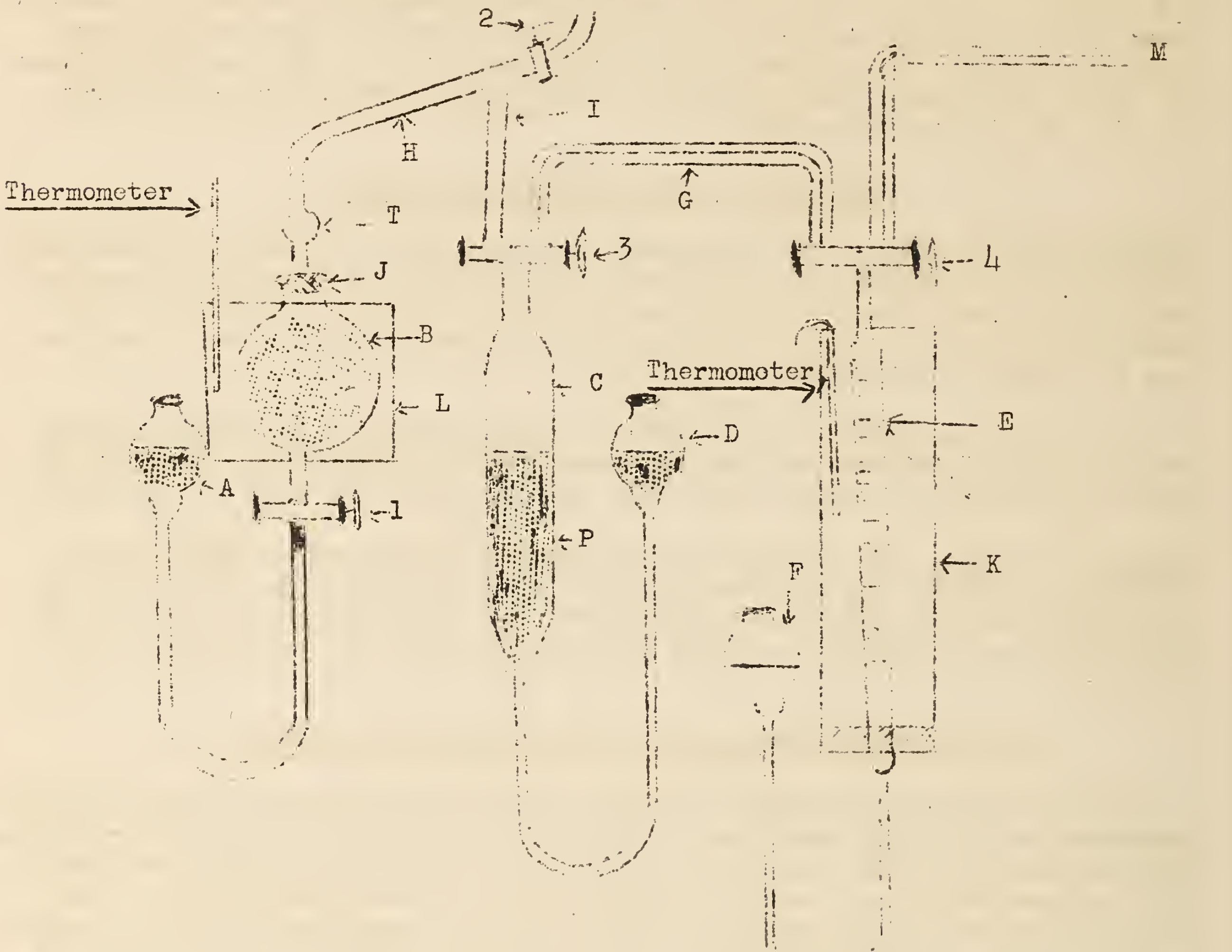

FIGURE 1. Gas analysis Apparatus

A. 500 cc. leveling bulb
B. 1000 cc. flask
C. Mercury pump, 150 cc. capacity, with three-way stopcock
D. 500 cc. leveling bulb
E. Gas buret, 50 cc. capacity, with three-way stopcock
F. 500 cc. leveling bulb
G. Capillary tubing, 1.5 mm. bore
H. Glass tubing, 1.5 mm. bore
K. Water jacket
L. Water bath
M. Connection for absorption pipets
T. Trap, 5-cm. diameter

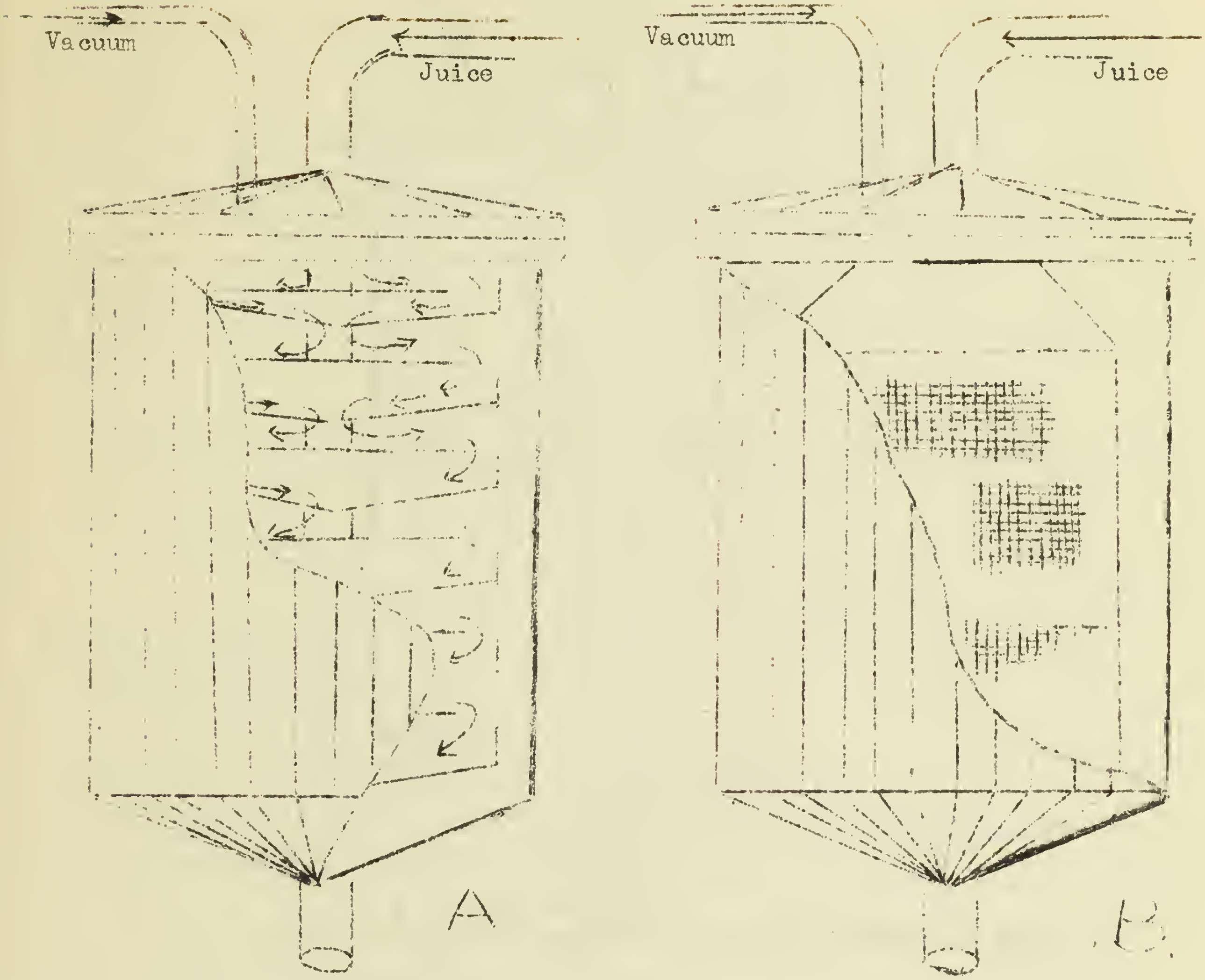

FIGURE 2.
Types of Deaerators used for Citrus Juices in Florida.

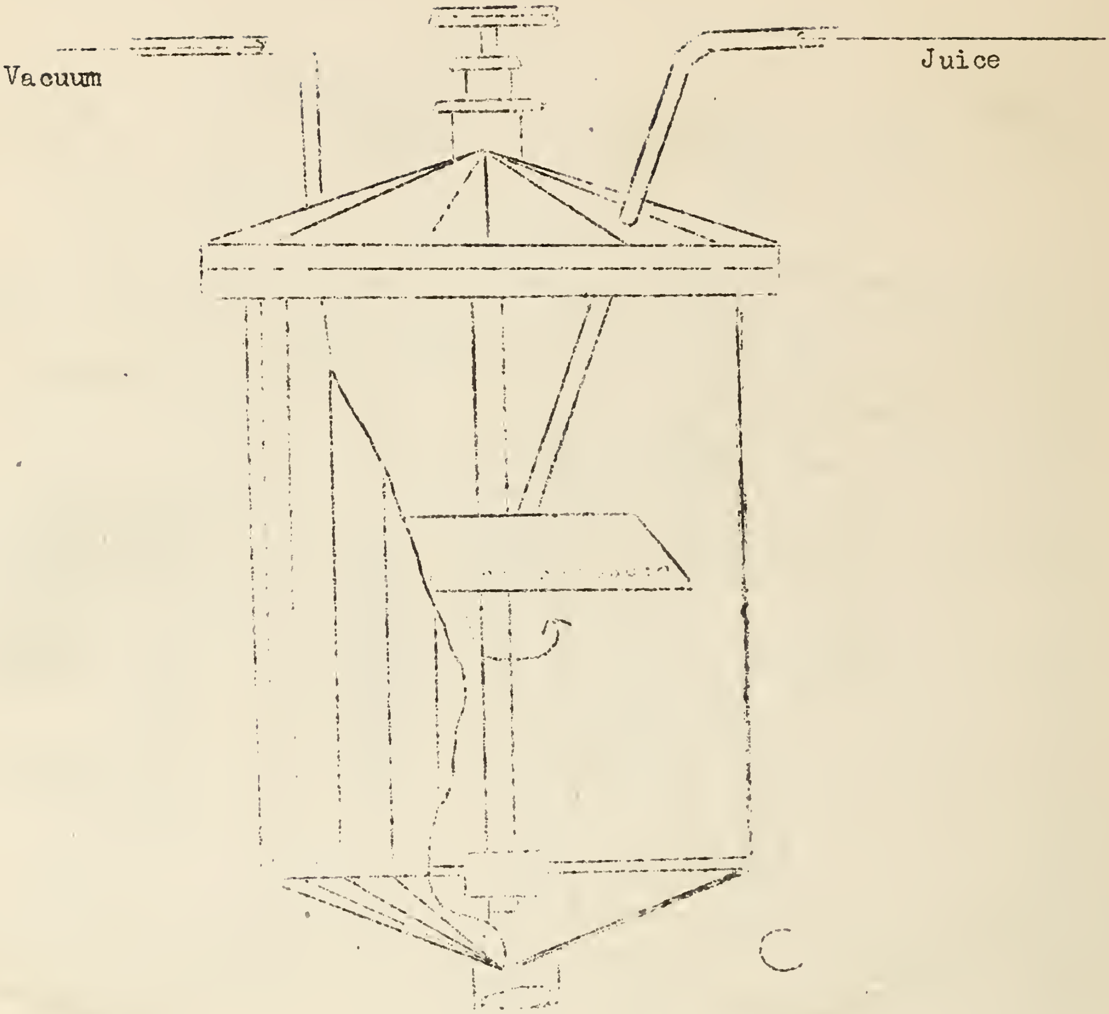

FIGURE 2. (cont'd.)
Types of Deaerators used for Citrus Juices in Florida.

Table II. Efficiency of Deaerators

Type	Tot. Gas	CO_2	O_2	N_2	Vacuum[a]	Approx. Reduction in O_2 %
	cc/liter at 0° (760 mm)				In (mm)	
Sprayed into flask (4)	19.2	17.4	0.12	1.7	27(686)	96.6
Raw juice	48.2	31.1	3.47	13.6	....	
Held in flask under vacuum	35.5	28.6	0.68	6.2	27(686)	73.1
Raw juice	54.9	38.7	2.52	13.7	....	
Centrifugal deaerator, lab. model	13.7	11.5	0.12	2.1	27(686)	95.3
Raw juice	54.9	38.7	2.52	13.7	....	
Plate type, commercial (Fig. 2A)	18.2	8.1	2.37	7.7	29(737)	43.3
Raw Juice	36.7	22.3	4.17	10.2	....	
Centrifugal type, commercial (Fig. 2C)	4.0	3.0	0.09	0.9	29(737)	97.2
Raw Juice	42.8	28.2	3.24	11.3	....	
Perforated cylinder, com. type (Fig. 2B)	17.2	8.1	2.39	6.7	25(635)	48.8
Raw juice	32.9	17.2	4.67	11.0	....	

[a] Measured with a Bourdon gage.

Table III. Juice Flow in Plate and Centrifugal Types of Deaerators

Type of Deaerator	Rate of Flow per min.		Flow per Min.		Oxygen in Deaerated Juice cc/L.
	Gal.	Liters	Gal/ sq.ft	L./ sq m	
Sprayed into flask (6)	0.61	2.3	0.23	9.4	0.12
Plate type (Fig. 2A)(4)	13.7	51.8	0.88	35.8	2.37
Perforated cylinder (Fig. 2B)	16.7	63.1	1.47	59.7	2.39
Centrifugal deaerator, lab. model	1.3	5.1	a	a	0.12
Centrifugal deaerator, commercial	10.0	37.8	a	a	0.09

[a] Deaeration does not depend upon surface area because the gas bubbles are fractured by impinging violently against the interior of the deaerator.

Table IV. Centrifugal Deaeration of Citrus Juices at Different Temperatures[a]

Temp. of Juice °C.	Total Gas	CO_2	O_2	N_2	Approx. Reduction in Oxygen, %
	Cc/liter at 0°C. (760 mm.)				
Raw	54.6	38.1	2.87	13.6	.....
6	28.4	21.5	1.06	5.8	63.1
13	22.1	18.3	0.35	3.4	87.8
16	21.9	17.9	0.12	3.8	95.9
26	15.8	13.9	0.12	1.5	95.9

[a] Vacuum constant at 27 inches (686 mm) or 3 inches (76.2 mm) absolute pressure.

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Table V. Centrifugal Deaeration of Citrus Juices at Different Vacuums[a]

Vacuum In. (mm.)	Absolute Pressure In. (mm)	Total Gas cc/liter at 0° C. (760 mm)	CO_2	O_2	N_2	Approx. Reduction in Oxygen, %
Raw juice		54.9	38.7	2.52	13.7	......
15 (381)	15 (381)	26.6	17.8	2.10	6.7	16.7
20 (508)	10 (254)	22.6	14.1	1.88	6.6	25.4
25 (635)	5 (127)	16.7	14.8	0.09	1.8	96.6
27 (686)	3 (76)	15.8	13.9	0.12	1.5	95.3

[a] Temperature constant at 16°C. (61°F.).

Table VI. Gases in Citrus Juices at Various Steps of Commercial Processing

Steps in Juice Processing	Tot.Gas Cc./liter at 0° C. (760 mm)	CO_2	O_2	N_2	Approx. Ratio O_2/N_2
		Plant 1			
Screened	36.7	22.3	4.17	10.2	0.409
In tank after sweetening	28.3	16.2	3.64	8.5	0.428
Deaerated (plate-type deaeration)	18.2	8.1	2.37	7.7	0.308
Pasteurized, canned	11.4	8.8	0.00	3.0	....
		Plant 2			
After screening and sweetening	35.6	22.3	2.46	10.7	0.230
Pasteurized, canned	25.9	21.6	0.00	4.2	....
		Plant 3			
After screening and sweetening	28.5	14.7	4.02	9.7	0.414
After vacuum sealing	23.2	14.2	2.47	6.5	0.380
Processed, canned	19.6	14.1	0.11	5.4	0.020
		Plant 4			
Reamed	42.8	28.2	3.24	11.3	0.287
Deaerated (centrifugal deaerator)	4.0	3.0	0.09	0.9	0.010
Pasteurized, canned	6.6	2.0	0.67	3.9	0.172

Table VII. Disappearance of Oxygen in Orange Juice

	Tot.Gas Cc/liter at 0°C. (760 mm)	CO_2	O_2	N_2	Approx. Reduction in Oxygen, %
Fresh juice	39.4	21.4	3.15	9.3	....
After standing in sealed (unprocessed) cans at room temp. 2 hr.	34.2	22.9	2.29	9.0	27.3
Fresh juice	23.2	14.2	2.47	6.5	....
After processing at 180° C. for 30 min. in can.	19.6	14.1	0.11	5.4	95.5
Fresh juice	44.2	29.1	3.40	11.7	....
After standing for 2 hr. in stoppered flask at room temp.	46.0	31.4	2.80	11.8	17.7